7/1/85
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ENVIRONMENTAL RESEARCH & TECHNOLOGY INC

MEMORANDUM

TO:	John Craun	**MEMO NO.:**	101-WGW-036
FROM:	Gary Wilson	**FILE:**	0005-354
SUBJECT:	Reagent Blank - RTCC St. Louis Park	**DATE:**	July 1, 1985

Shown on Table 1 are the concentrations of PAH for the reagent blank analyzed with the three water samples submitted from RTCC - St. Louis Park. Reagent blanks measure the presence of laboratory contamination whether as residual glassware contamination, sample cross-contamination, or reagent/standard contamination. Depending on the nature of the samples being analyzed, specific lot of reagents, etc., blanks vary from sample set to sample set. However, they should generally be low. Final sample concentrations reflect a mathematical correction for the method blank for a specific set of samples. Therefore, all samples, including the field blank, would have been corrected for the reagent blank shown on Table 1.

In response to request from EPA Region VIII, we have performed studies specific to the GC/MS SIN method. The first is replicate analyses of distilled water blanks. Shown on Table 1 are the average and standard deviation of PAH concentrations for eighteen (18) replicates of laboratory blanks, at an instrument detection limit of 2 parts per trillion.

Secondly, EPA has a method for determining method detection limit (MDL) which takes into account blank variability and recovery variability. Distilled water blanks are fortified at 1-5 times the expected detection limit. The blanks are subjected to the entire analytical procedure. The concentration mean and standard deviation are calculated and the MDL is defined as 2.998 times the standard deviation. This seems a harsh criteria at part per trillion levels (it was developed for analyses at typical ppb levels) since the variability is geater at lower concentration. However, we have performed the study using eight (8) distilled water samples spiked at 10 ppt. The results are shown on Table 2. For some EPA work we will be held to these MDLs even though our instrumental detection limit is less than two (2) ppt. We are presently doing similar calculations for the common heterocycles and some additional PAH.

As a result of increasing background levels in our laboratory, as indicated by laboratory blank analyses, ERT has taken special steps to decrease laboratory contamination. The blank study indicates that we have successfully controlled our

laboratory background levels.

The RTCC lab blank is higher than we would now want to see. However, at the time it was extracted and analyzed, we had not identified and made the above-mentioned changes. The purpose of the lab blank is to define the level of background contamination. This level changes, especially at ppt concentrations, as laboratory conditions change. We feel that in the future, using our latest precautions, we will see less fluctuation and be able to maintain a consistantly low level.

Still, the RTCC lab blank is not unreasonably high, nor does it account for the levels of PAH in the field blanks. We generally see higher field blanks whenever samples of high concentration are also being sampled. The field blank is a measure of field and shipping contamination in the same way that a laboratory blank measures the laboratory background contamination. As we discussed when we talked last week, it would be wise to look at field notes to determine where the field blanks were transferred and in which cooler the bottles were shipped.

I hope this information is helpful. Please call me if you have any questions.

Gary (Ka)

WGW/rn
Attachments

TABLE 1

LABORATORY BLANKS

	RTCC LAB BLANK	BLANK STUDY (N=18) MEAN	STD. DEV.
Naphthalene	110	11	3.9
Acenaphthene	BDL	BDL	0
Acenaphthylene	BDL	BDL	0
Fluorene	BDL	BDL	0
Phenanthrene/ Anthracene	21	3	1.9
Fluroanthene	BDL	BDL	0
Pyrene	12	BDL	0
Benz(a)anthracene/ Chrysene	15	BDL	0
Benz(a)pyrene	10	BDL	0
Benzofluoranthenes	10	BDL	0
Indeno(1,2,3-cd)pyrene	10	BDL	0
Benzo(ghi)perylene	BDL	BDL	0
Dibenz(ah)anthracene	BDL	BDL	0
Indene	BDL		
Indole/2,3-dihydroindene	77		

	RTCC Lab Blank
2,3-benzofuran	59
Benzo(b)thiophene	35
2-Methyl Naphthalene	5
1-Methyl Naphthalene	BDL
Carbazole	13
Acridine	13
Dibenzo Thiophene	BDL
Perylene	BDL
Quinoline	17
Biphenyl	160

BDL = Below Detection Limit

Results in part per trillion (ppt) (Ng/l)

TABLE 2

EPA DETECTION LIMIT STUDY

	Mean	Standard Deviation	Variance	MDL*	95% Confidence Limits
Naphthalene	1.8	2.9	8.4	9	6-17
Acenaphthylene	4.2	0.8	0.7	2	1-7
Acenaphthene	5.9	1.2	1.4	3	2-6
Fluorene	6.0	2.5	6.5	8	5-15
Phenanthrene/ Anthracene	15	3.3	11	10	7-19
Fluoranthene	9.6	1.3	1.7	4	3-8
Pyrene	7.9	1.4	1.8	4	3-8
Benz(a)anthracene/ Chrysene	16	2.4	5.6	7	5-13
Benzofluoranthenes	14	2.2	4.9	7	5-13
Benz(a)pyrene	7.6	1.3	1.7	4	3-8
Indeno(1,2,3-cd) pyrene	7.0	1.1	1.2	3	2-6
Dibenz(ah)anthracene	7.0	1.6	2.5	5	3-10
Benzo(ghi)perylene	6.8	1.3	1.7	4	3-8

Results in ppt

MDL = Method Detection Limit

ENVIRONMENTAL RESEARCH & TECHNOLOGY INC

MEMORANDUM

TO:	John Craun	**MEMO NO.:**	100-WGW-033
FROM:	Gary Wilson	**FILE:**	0005-354
SUBJECT:	Analyses for RTCC Wells at SLP	**DATE:**	June 13, 1985

Shown on Table 1 are the analytical results for the analysis of PAH and selected heterocycles at trace levels. This is the completed version of the table given you on 31 January 1985.

If you have any questions, please contact me.

WGW/rn

cc: J. Mastone
Lee Keller, Dorsey & Whitney
Carl Lesher, RTCC

TABLE 1: ANALYTICAL RESULTS

Results in ppt (ng/l)

Compound	Detection Limit	25002 Well W-23	25011 Well SLP-4	25003 Field Blank
Naphthalene	2	874,000	110	79
Acenaphthene	2	16,600	BDL	BDL
Acenaphthylene	2	29,400	320	19
Fluorene	2	51,200	13	35
Phenanthrene/ Anthracene	2	42,600	163	102
Fluoranthene	2	4,240	51	73
Pyrene	2	5,710	43	53
Benz(a)anthracene/ Chrysene	2	1,810	8	22
Benz(a)pyrene	4	67	6	12
Benzo Fluoranthenes	4	51	5	14
Indeno(1,2,3-cd)pyrene	4	12	BDL	9
Benzo(g,h,i)perylene	4	10	BDL	BDL
Dibenz(a,h)anthracene	4	13	BDL	10

TABLE 1 (cont.)

Compound	Detection Limit	25002 Well W-23	25011 Well SLP-4	25003 Field Blank
Indene	4	98700	75	11
Indole /				
2,3-dihydroindene	10	92300	560	110
2,3-benzofuran	4	10500	28	45
Quinoline	4	920	BDL	82
Benzo(b)thiophene	4	41600	170	BDL
2-methyl naphthalene	4	8540	73	12
1-methyl naphthalene	6	190,000	100	15
Biphenyl *	50	20,200	BDL	2590
Carbazole	4	12,400	71	BDL
Acridine	4	1660	BDL	BDL
Dibenzothiophene	4	4070	BDL	BDL
Perylene	4	BDL	BDL	BDL
SURROGATES, % RECOVERY				
D-8 Naphthalene		SI	24	89
D-10 Fluorene		90	37	220
D-12 Chrysene		93	14	108

SI = Sample Interference
BDL = Below Detection Limit

* Detection Limit is high because of contamination interferences.

ERT
A COMSAT COMPANY
ENVIRONMENTAL RESEARCH & TECHNOLOGY, INC.
PORTER BUILDING, 10th FLOOR, 601 GRANT STREET, PITTSBURGH, PENNSYLVANIA 15219, (412) 261-2910

ERT Project No: B690
July 10, 1985

Mr. Carl F. Lesher
Reilly Tar & Chemical Corporation
1510 Market Square Center
151 N. Delaware Avenue
Indianapolis, IN 46204

Re: Samples Split by ERT and TCT

Dear Carl:

Enclosed are various items related to analyses by ERT and TCT of split samples of W23 and SLP4 collected on December 10 and 11, 1984, respectively. First are copies of ERT's and TCT's lab reports, which you should have seen already. Second is a recent memo by Gary Wilson on the lab blank for ERT's work. Gary prepared this in response to our concerns over the high field blank results obtained by ERT compared to SLP4 results. Finally, I have prepared a table summarizing ERT's and TCT's field blank and split sample results.

A few comments are in order regarding these items. First, ERT's and TCT's results do not compare very well, with ERT's results generally being much higher (see Table 1). I suspect that this is due to the long delay (over 3 months) between sample collection and analysis by TCT, versus about half this time for ERT's work. You will recall that TCT's data for Calgon's ACT test feed showed apparent sample degradation over the 42-day test, particularly for the smaller PAH. Also note that by far the biggest discrepancy between ERT and TCT results is for 2-ring PAH in W23 samples, where volatilization losses during a long storage period could be a problem. In contrast, both lab's W23 results for 3-ring and 4-ring PAH agree within 50% and compare favorably with ERT's results for the March 1985 W23 sample collected for odor testing (see Table 1). W23 sample results by the University of Iowa also indicate that TCT's results for 2-ring PAH are suspiciously low (Table 1).

Second, the field blank results reported by ERT are indeed higher than ERT's results for SLP4 (see Wilson's 6/13/85 memo and Table 1). However, ERT's field blank was collected at W23, and TCT's results also show the W23 field blank being higher than the SLP4 sample. Moreover, TCT's results for the W23 field blank are 5 times higher than their SLP4 blank results. This fits with reports I have heard from John Ryan and Gary Wilson on ERT sampling and analysis work at Burlington Northern sites, where field blanks transferred near highly contaminated wells do show definite contamination (see Wilson's 7/1/85 memo).

I hope this letter and attachments answer your questions about the results of the ERT/TCT sample splitting experiment. While the test results are somewhat disappointing, I think they are explainable and generally consistent with earlier results at these wells. Let me know if you have any questions or comments.

Sincerely,

John C. Craun

JCC:JLH
Attachments

cc: R.A. Upham (TCT)
W.G. Wilson (ERT)

P.S. I checked with Upham and Wilson on sample extraction and analysis dates and got the following information:

Action	ERT	TCT
collection	12/10&11/84	12/10&11/84
extraction	12/14/84	12/13-17/84
injection	1/22/85 (PAH) 2/28/85 (heteros)	3/19-21/85

TABLE 1

COMPARISON OF ERT AND TCT RESULTS FOR SLP4 AND W23 SPLIT SAMPLES

Sample	Lab	Total Concentration of Compounds Analyzed by Both Laboratories, ng/l(e) 2-Ring(a)	3-Ring(b)	4-Ring(c)	5-Ring(d)	Total
December, 1985 Splits:						
Lab Blank	ERT	463	47	27	30	567
W23 (i)	ERT	1,336,760	153,860	11,760	153	1,502,533
	TCT	75,398	103,718	16,487	415	196,018
W23 Field Blank	ERT	2,944	156	148	45	3,293
	TCT	1,045	76	53	ND(g)	1,174
SLP4	ERT	1,116	567	102	11	1,796
	TCT	233	75	37	3	348
SLP4 Field Blank	TCT	182	15	39	3	239
Other W23 Samples:						
3/85 Odor Test Sample(i)	ERT(j)	211,120	121,590	19,900	5,960	358,570
10/83 after air blasting:						
1/2 hour pumping	Iowa(k)	843,000(h)	334,000	30,000	ND(f)	1,207,000
8 hours pumping	Iowa(k)	707,000(h)	260,000	17,000	ND(f)	984,000
Other SLP4 Samples:						
9/82	MRC(l)	-(m)	142	16	2	-(m)
9/82	CH2MHill(l)	288/306(h)	234/241	ND(g)	ND(g)	522/547
11/82	CH2MHill(l)	431/280(h)	373/256	ND(g)	ND(g)	711/536

Notes:

(a) Indene, Indole, 2,3-Dihydroindene 2,3-Benzofuran, Quinoline, Benzo(b)thiophene, Naphthalene, 1-Methylnaphthalene, 2-Methylnaphthalene, Biphenyl.

(b) Acenaphthene, Acenaphthylene, Fluorene, Phenanthrene, Anthracene, Acridine, Carbazole.

(c) Fluoranthene, Pyrene, Benz(a)anthracene, Chrysene.

(d) Benzo(a)pyrene, Benzofluoranthenes, Indeno(1,2,3-cd)pyrene, Benzo(ghi)perylene, Dibenz(a,h)anthracene, Perylene.

(e) In addition, ERT alone analyzed for Dibenzothiophene and TCT alone analyzed for Dibenzofuran, Phenanthridine and Benzo(e)pyrene.

(f) At 1,000 ng/l detection limit per compound.

(g) At low ng/l detection limits.

(h) Does not include analysis for 2,3-benzofuran.

(i) Sample collcted after about 3 1/2 hours of pumping.

(j) From ERT's 5/30/85 report "PAH Threshold Odor Determination in St. Louis Park Municipal Supply Water".

(k) From Table 4 of 11/10/83 Josephson to Simonett letter (doc. no. 9626753-57).

(l) From CH2M Hill Tech Memo D, Attachment D4. CH2M Hill results are for duplicate samples.

(m) MRC only analyzed for 4 of the compounds listed in note a.

twin city testing
and engineering laboratory, inc.
662 CROMWELL AVENUE
ST PAUL MN 55114
PHONE 612/645 3601

REPORT OF: PAH ANALYSIS BY GCMS

PROJECT: REILLY TAR & CHEMICAL

DATE: March 26, 1985

REPORTED TO: Dorsey & Whitney
Attn: Mr Schwartzbauer
200 First Bank Place East
Minneapolis, MN 55402

FURNISHED BY: 1-Reilly Tar & Chem Corp
Attn: Paul Rivers

COPIES TO: 1-ERT
Attn: Mr John Craun

LABORATORY No. 2A-3077

INTRODUCTION

This report presents the results of PAH analysis of water samples. These samples were collected at the wells indicated below. The work was requested by Mr John Craun of ERT and authorized by Mr Edward Schwartzbauer of Dorsey and Whitney.

SAMPLE IDENTIFICATION

TCT Number	Identification	
9432	Well 48 Methodist Hospital	Acurex Split
9433	Well 48 Methodist Hospital (Duplicate)	Acurex Split
Meth Blank	Field Blank Methodist Hospital	Acurex Split
9689	Field Blank American Mutual Hardware	
9691	American Mutual Hardware	
10308	SLP Well 6	Acurex Split
10309	SLP Well 6 Field Blank	Acurex Split
10337	Well 23 Blank	Acurex Split
10338	Well 23	Acurex Split
10366	SLP - Well 4	Acurex Split
10367	SLP - Well 4 Field Blank	Acurex Split
10419	SLP - Well 40 Minnesota Rubber	Acurex Split
10420	SLP - Well 40 Minnesota Rubber	Bottle Blank Acurex Split

METHODOLOGY

The one liter sample was spiked with surrogate standards and the pH of the water was adjusted to 11 with sodium hydroxide. The sample was extracted with 3 x 60 uL portions of Burdick and Jackson methylene chloride. The extract was passed through a column of anhydrous sodium sulfate and concentrated to 0.5 mL in a Kuderna Danish apparatus. The concentrated extract was transferred to a calibrated 2 mL vial. The solution was further concentrated to 1 mL with dry nitrogen, 150 uL of toluene was added and concentration was continued to 0.1 mL.

A 50 uL portion of the extract was diluted with a 50 uL portion of d_{10} phenanthrene in toluene. A 1 uL portion of this solution was injected directly onto a J & W 30 meter DB5 quartz capillary column with a J & W on-column injector. The column was temperature programmed and the column exit was coupled directly to the source of a VG 7070E mass spectrometer. The mass spectrometer was operated at 2000 resolution with a 1000 ppm wide flat-topped peak. Data collection was by selected ion recording using the VG 2035 data system.

twin city testing
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REPORT OF: PAH ANALYSIS BY GCMS

DATE: March 26, 1985

LABORATORY No. 2A-3077

DISCUSSION

The results of these analyses are contained in Tables I and II. Table I contains data from selected ion recording (SIR) analysis. All samples except those for SLP 40 and Well 23 were extracted with the contaminated lots of methylene chloride (see TCT Report Number 2A-3301 for a complete discussion of this problem). SLP 40 and Well 23 were extracted with amylene preserved methylene chloride. Well 23 and its field blank were analyzed by full scan GCMS thus resulting in slightly higher detection limits.

REMARKS

All extractions will be held for a period of one month from the date of completion.

TWIN CITY TESTING AND
ENGINEERING LABORATORY INC

Roger A Upham, Ph.D
Senior Consultant

William F Welbes
Manager-Organic Chemistry

RAU/WFW/ms

TABLE I

Reilly Tar and Chemical P.A.H. Analysis

	Meth. Hosp. 9432	Meth. Hosp. (Dup.) 9433	Meth. Hosp. Field Blank	Am. Hardware Mutual 9691	9689 Blank	SLP6 10308	10309 Blank	SLP4 10366	10367 Field Blank	Minn. Rubber Well 40 10419	10420 Field Blank	Lower Detectable Limit
2,3 Benzofuran	ND	4.8	1.8	ND	1.4	1.7	1.2	5.5	ND	ND	1.6	1.0
2,3 Dihydroindene	60	28	5.7	1.8	1.3	7.4	2.6	12	3.4	1.2	3.2	1.0
Indene	6.5	11	2.1	1.3	ND	4.7	1.0	8.2	2.2	ND	2.0	1.0
Naphthalene	32	30	25	15	12	66	40	49	51	2.3	26	1.0
Benzo (b) Thiophene	11	25	ND	ND	ND	2.7	ND	21	ND	ND	ND	1.0
Quinoline	ND	3.1	2.9	ND	ND	ND	1.2	1.5	1.3	ND	ND	1.0
Indole	ND	2.8	*1	ND	13	1.1	ND	2.8	2.3	ND	ND	1.0
2-Me-Naphthalene	26	20	26	37	48	63	40	45	42	7.9	19.6	1.0
1 Me-Naphthalene	44	33	43	74	82	84	79	80	75	16	34	1.0
Biphenyl	30	28	2.0	1.4	2.2	3.6	3.3	7.9	5.0	3.9	1.3	1.0
Acenaphthylene	45	60	ND	ND	ND	1.2	ND	1.8	ND	18	ND	1.0
Acenaphthene	103	119	1.4	11	1.8	4.9	2.1	30	3.8	54	ND	1.0
Dibenzofuran	11	18	2.6	ND	1.2	1.2	ND	1.5	1.2	ND	ND	1.0
Fluorene	179	244	2.4	1.4	2.8	3.1	2.1	2.6	4.3	85	2.7	1.0
Phenanthrene	13	14	15	4.8	12	6.6	6.4	14	7.0	6.9	4.6	1.0
Anthracene	8.6	8.4	2.1	ND	ND	1.4	ND	2.3	ND	6.0	ND	1.0
Acridine	9.2	16	3.8	1.6	ND	ND	ND	ND	ND	4.4	ND	1.0
Phenanthridine	1.3	2.6	4.8	ND	ND	ND	ND	ND	1.2	ND	ND	1.0
Carbazole	1.6	2.5	1.3	ND	1.2	1.1	ND	24	ND	1.1	ND	1.0
Fluoranthene	142	172	27	5.0	17	4.3	5.3	4.8	4.1	46	2.8	1.0
Pyrene	121	133	12	17	16	4.0	6.7	4.3	3.2	69	2.5	1.0
Benzo (a) anthracene	11	15	ND	3.1	27	2.5	5.2	28	32	5.8	1.3	1.0
Chrysene	5.6	7.8	20	2.3		1.2	3.3			3.4	1.3	1.0
Benzo (b & k) fluoranthene	1.6	3.0	8.6	38	5.9	1.1	5.8	1.6	2.2	1.0	ND	5.0
Benzo (e) pyrene	ND	2.0	4.2	ND	2.0	ND	2.7	1.2	1.0	ND	ND	5.0
Benzo (a) pyrene	ND	2.0	3.7	ND	ND	ND	3.3	ND	ND	ND	ND	5.0
Perylene	ND	ND	2.0	ND	ND*1	ND	ND	ND	ND	ND	ND	5.0
Indeno (1,2,3,cd) pyrene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5.0
Dibenzo (a,h) anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	5.0
Benzo (a,h,i) perylene	ND	1.5	2.7	ND	ND	ND	ND	ND	ND	ND	ND	5.0

All concentrations are in ng/L (parts per trillion)
*Interference
ND - Not Detected

TABLE I

Reilly Tar and Chemical P.A.H. Analysis

- Continued -

	Meth. Hosp. 9432	Meth. Hosp. (Dup.) 9433	Meth. Hosp. Field Blank	Am. Hardware Mutual 9691	9689 Blank	SLP6 10308	10309 Blank	SLP4 10366	10367 Field Blank	Minn. Rubber Well 40 10419	10420 Field Blank
Surrogate Recovery:											
Decafluorobiphenyl, % (recovery)	7.7	9.7	6.8	28.8	19.2	28.8	24.2	21.9	42.5	2.9	5.5
D8 Naphthalene, % (recovery)	10.5	8.9	6.6	26.5	13.9	25.5	25.5	20.9	32.0	0.4	3.8
D7 Quinoline, % (recovery)	12.8	13.3	22.7	36.0	24.0	36.0	33.0	30	44	7.2	10.4
D12 Chrysene, % (recovery)	39.9	38.9	5.8	106.7	81.5	61.8	106.7	47.8	120.8	73.0	56.2

TABLE II

Reilly Tar and Chemical P.A.H. Analysis

	Well 23 10338	10337 Blank	Lower Detectable Limit
2,3 Benzofuran	937	ND	5
2,3 Dihydroindene	17140	14.5	5
Indene	11451	7.3	5
Naphthalene	356	310	5
Benzo (b) Thiophene	277	ND	5
Quinoline	ND	ND	5
Indole	ND	ND	5
2-Me-Naphthalene	1076	310	5
1-Me-Naphthalene	39079	403	5
Biphenyl	5082	ND	5
Acenaphthylene	5689	ND	5
Acenaphthene	26822	nd	5
Dibenzofuran	17681	ND	5
Fluorene	37244	ND	5
Phenanthrene	20123	76	5
Anthracene	5590	ND	5
Acridine	1313	ND	5
Phenanthridine	ND	ND	5
Carbazole	6937	ND	5
Fluoranthene	9088	26.4	5
Pyrene	6633	26.4	5
Benzo (a) anthracene	462	ND	5
Chrysene	304	ND	5
Benzo (b & k) fluoranthene	297	ND	25
Benzo (e) pyrene	ND	ND	25
Benzo (a) pyrene	ND	ND	25
Perylene	ND	ND	25
Indeno (1,2,3,cd) pyrene	59	ND	25
Dibenzo (a,h) anthracene	ND	ND	25
Benzo (a,h,i) perylene	59	ND	25
Surrogate Recovery:			
Decafluorobiphenyl, % (recovery)	24.1	25.3	
D8 Naphthalene, % (recovery)	43.2	42.9	
D7 Quinoline, % (recovery)	33.0	33.2	
D12 Chrysene, % (recovery)	100	100.8	

All concentrations are in ng/L (parts per trillion)
ND - Not Detected